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IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicants:	Masayuki YAMANA et al.	Conf.:	6781
Appl. No.:	09/355,673	Groupt:	1713
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Title:	METHOD FOR TREATMENT OF TEXTILE AND TREATED TEXTILE		

DECLARATION UNDER 37 C.F.R. § 1.132

I, Masato KASHIWAGI, a citizen of Japan, residing at c/o Yodogawa Works of Daikin Industries Ltd., 1-1, Nishihitotsuya, Settsu-shi, Osaka-fu, Japan declare and say as follows:

1. I am one of the co-inventors of the above-identified application.

2. I graduated from Osaka City University, Faculty of Engineering, Department of Applied Chemistry in March 1984. I graduated from graduate school of Osaka City University, and received a master's degree in Engineering in March 1986.

3. Since April 1986 up to the present time, I have been employed by Daikin Industries Ltd. and engaged in research works on the development of the synthesis and application of fluorine-containing compounds.

4. I have performed the following experiments and beg to submit herewith the exact report thereon.

The following Experiment 1 and Comparative Experiment 1 were conducted.

Water repellency and oil repellency shown in the following Experiments are expressed by the following evaluations.

Water repellency is expressed in terms of water repellency No. (see Table 1 below) by a spray method in accordance with JIS (Japanese Industrial Standard) L-1092.

Oil repellency is expressed in terms of oil repellency No. by observing the state whether the drop can be maintained on the cloth for 30 seconds after one drop (about 5 mm in diameter) of a test solution shown in Table 2 below is placed on a sample cloth (AATCC TM118-1992).

Table 1

Water repellency No.	State
100	No wet on the surface
90	Slight wet on the surface
80	Partial wet on the surface
70	Wet on the surface
50	Wet on the whole surface
0	Complete wet on the front and back surfaces

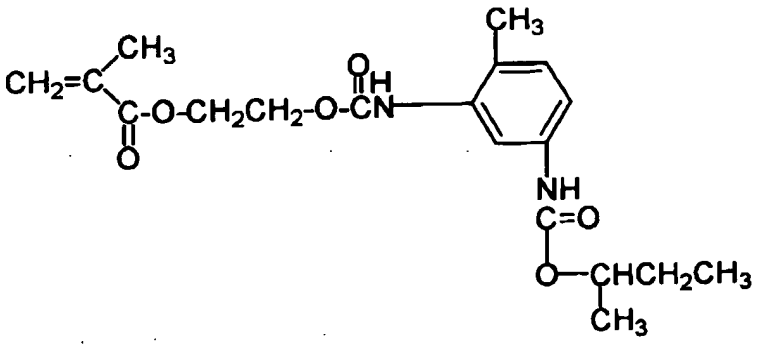
Table 2

Oil repellency No.	Test solution	Surface tension (dyne/cm, 25℃)
8	n-Heptane	20.0
7	n-Octane	21.3
6	n-Decane	23.5
5	n-Dodecane	25.0
4	n-Tetradecane	26.7
3	n-Hexadecane	27.3
2	Mixture of n-hexadecane/nujol (35/65)	29.6
1	Nujol	31.2
0	inferior to 1	-

Experiment 1

This Experiment 1 evaluates the water- and oil-repellent which is the same as Preparative Experiment 17 in the present Description.

152 g of CH_2=CH-C(=O)O-$CH_2CH_2C_8F_{17}$ (a fluorine-containing monomer), 85 g of a 66.7% di-n-butyl adipate solution of a urethane bond-containing monomer:



40 g of lauryl methacrylate, 7.5 g of 3-chloro-2-hydroxypropyl methacrylate, 5 g of glucosyl ethyl methacrylate (50% aqueous solution), 12.5 g of lauryl mercaptan, 3.5 g of di hardened beef tallow alkyl dimethyl ammonium chloride (active ingredient of 75%), 1,250 g of deionized water; and 1 g of di-n-butyl adipate, 86 g of butyl carbitol acetate and 84.5 g of dipropylene glycol monomethyl ether as a film-forming auxiliary were charged and pre-emulsified by using a high pressure homogenizer. This emulsion was transferred to a flask equipped with a stirrer, a thermometer and a reflux condenser, nitrogen purge was conduct d sufficiently at 60°C, and then 0.5 g of 2,2'-azobis(2-amidinopropan)dihydrochloride was add d to initiate the polym rization. At 5 hours aft r the initiation of polymerization, it was confirmed that 99% of the

fluorine-containing monomer [CH_2=CH-C(=O)O-$CH_2CH_2C_8F_{17}$] had reacted by gas chromatography and a water- and oil-repellent (in the form of a latex) having the solid content of 15% was obtained.

The water- and oil-repellent was diluted with deionized water so as to have a solid content of 2% and this liquid was uniformly sprayed on a cotton cloth, a polyester/cotton(65/35) cloth, a polyester cloth and a nylon cloth so that a wet pickup was 100%. Spray was carried out using a hand spray (a trigger type container). Then, these cloths were dried at room temperature for 24 hours. The water and oil repellency test were carried out for these treated cloths. The test results are shown in Table A.

Comparative Experiment 1

This Comparative Experiment 1 evaluates a water- and oil repellent obtained in Examples 3 of US Patent No. 5,068,295.

A monomer consisting of 100 g of CH_2=C(CH_3)-C(=O)O-$CH_2CH_2C_8F_{17}$, 30 g of CH_2=C(CH_3)-C(=O)O-$(CH_2)_3$-Si$(CH_3)_2$-[OSi$(CH_3)_2]_{19}$-CH_3, and 30 g of 2-hydroxyethyl acrylate/tolylene diisocyanate/acetoxime adduct (molar ratio 1:1:1); 100 g of acetone; 3 g of a non-ionic emulsifier (polyoxyethylene(23)cetyl ether); 3 g of dimethyloctadecylamine acetate; and 565 g of deionized water were charged into a reactor. The reactor was sufficiently purged with nitrogen, and then 0.8 g of azoisobutylamidine dihydrochloride was added under the nitrogen atmosphere to initiate the polymerization. The polymerization was conducted at 65°C for 15 hours under the nitrogen atmosphere to give a water- and oil-repellent (in the form of a latex) having a solid content of 20% by weight.

The water- and oil-repellent was diluted with deionized water so as to have a solid content of 2% and this liquid was uniformly sprayed on a cotton cloth, a polyester/cotton(65/35) cloth, a polyester cloth and a nylon cloth so that

a wet pickup was 100%. Spray was carried out using a hand spray (a trigger type container). Then, these cloths were dried at room temperature for 24 hours. The water and oil repellency test were carried out for these treated cloths. The test results are shown in Table A.

Table A

		Experiment 1	Comparative Experiment 1
Water repellency	Cotton cloth	90	50
	Polyester/cotton (=65/35) cloth	80	50
	Polyester cloth	80	50
	Nylon cloth	90	50
Oil repellency	Cotton cloth	4	0
	Polyester/cotton (=65/35) cloth	5	1
	Polyester cloth	5	0
	Nylon cloth	6	1

Table A shows that the water- and oil-repellent of Experiment 1 (according to the present invention) imparts much better water repellency and oil repellency than Comparative Experiment 1 (according to US Patent No. 5,068,295), when the treated cloth is dried at room temperature without heating the treated cloth.

In the copolymer prepared in Experiment 1,

(a1) the fluorine-containing monomer: $CH_2=CH-C(=O)O-CH_2CH_2C_8F_{17}$ corresponds to the repeating unit (I) defined in the present claims,

(a2) the urethane bond-containing monomer corresponds to the repeating unit (II),

(a3) lauryl methacrylate corresponds to the repeating unit (III),

(a4) glucosyl ethyl methacrylate corresponds to the repeating unit (IV), and

(a5) 3-chloro-2-hydroxypropyl methacrylate corresponds to the repeating unit (IV).

That is, th copolymer in Experiment 1 has the r p ating units (I), (II), (III), (IV) and (V).

In the copolymer prepared in Comparative Experim nt 1,

(b1) $CH_2=C(CH_3)-C(=O)O-CH_2CH_2C_8F_{17}$ corresponds to the repeating unit (I) defined in the present claims,

(b2) 2-hydroxyethyl acrylate/tolylene diisocyanate/acetoxime adduct corresponds to the repeating unit (II), and

(b3) $CH_2=C(CH_3)-C(=O)O-(CH_2)_3-Si(CH_3)_2-[OSi(CH_3)_2]_{19}-CH_3$ may correspond to the repeating unit (III).

That is, the copolymer in Comparative Experiment 1 has the repeating units (I), (II) and (III) and lacks in both of the repeating units (IV) and (V).

In the present invention at least one of the repeating units (IV) and (V) is essential in addition to the repeating units (I), (II) and (III). Experiment 1 is included in the present invention and Comparative Experiment 1 is excluded from the present invention.

It is clear that the presence of the repeating units (IV) and (V) gives much better water repellency and oil repellency when Experiment 1 is compared with Comparative Experiment 1.

The undersigned declares further that all statements made herein of his own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under 18 U.S. Code 1001 and that such willful false statements may jeopardize the validity of this application or any patent issuing thereon.

Masato KASHIWAGI

Dated this day of June 2002